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Office of Environmental Measurement and Evaluation
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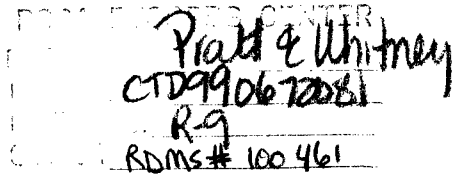

MEMORANDUM

DATE: June 20, 2002

SUBJ: Laboratory Technical Systems Audit Report
PCB Analysis
Premier Laboratory, LLC
Dayville, CT

FROM: Nora J. Conlon, Ph.D., QA Chemist
Ann R. Jefferies, QA Chemist
Quality Assurance Unit, OEME

TO: Kimberly Tisa
Pesticides, Toxics and Radiation Unit, OEP
Juan Perez, RCRA Facility Manager
RCRA Corrective Action Unit, OSRR

SCOPE

A technical systems audit (TSA) of Premier Laboratory was conducted by an EPA NE Quality Assurance TSA team on June 18, 2002. The TSA was performed to evaluate the procedures for PCB analysis, primarily in support of the Toxic Substance Control Act (TSCA). The criteria used to conduct the TSA were the laboratory Standard Operating Procedures (SOPs), the Premier Laboratory Quality Manual, Rev. 2.3, March 1, 2002 and general good laboratory practice. Participants in the TSA are listed below.

Premier Laboratory staff:

Ron Warila	Laboratory Director
Bob Stevenson	Quality Assurance/Quality Control Manager
Philip Rusconi	CEO
Robert Laferriere	General Manager
Victor LeClerc	Sample Custodian
Bill Mallory	Sample Custodian
Rich Warila	Organics Manager
Scott Lamitie	Sample Prep
Amanda Swider	Sample Prep
Weison Huang	Pesticide/PCB Analyst

EPA Quality Assurance TSA team:

Nora Conlon	Quality Assurance Chemist
Ann Jefferies	Quality Assurance Chemist

EXECUTIVE SUMMARY

Premier Laboratory was found to be operating in accordance with their SOPs, their Quality Manual and good laboratory practices for PCBs in aqueous and solid samples. The laboratory has a customized Laboratory Information Management System (LIMS) that minimizes chances for transcription error and allows expedited multiple data reviews. The staff was experienced, knowledgeable and cooperative throughout the TSA.

AUDIT PROCEDURES

The TSA team reviewed the Laboratory Quality Manual, Rev. 2.3, Effective Date: March 1, 2002, Method Detection Limit results for PCBs, and the most recent SOPs:

Pressurized Fluid Extraction, Method 3545, Rev. 1.0, October 19, 2001
Separatory Funnel Liquid-Liquid Extraction, Method 3510C, Rev. 1.2, April 3, 2002
Sulfuric Acid Cleanup for PCBs, Method 3665A, Rev. 1.1, October 19, 2002
Sulfur Cleanup, Method 3660B, Rev. 1.1, October 19, 2002 (2001?)
Polychlorinated Biphenyls by Gas Chromatography, SW-846 8082, Rev. 1.1, March 12, 2002
General Quality Assurance SOPs for Sample & Data Management.

The following laboratory systems were examined by the TSA team:

- Sample Receipt, Storage and Log-In
- Analytical Procedures for Solid and Aqueous PCB Samples
  - Sample Preparation
  - Standard Preparation
  - Calibration
  - Sample Analysis
  - Qualitative Identification
  - Quantitative Calculations
  - Quality Control Analysis, i.e., Blanks, Surrogates, Laboratory Control Samples, etc.
- Data Reduction, Review and Reporting
- Quality Assurance Program

FINDINGS

There were no findings that require corrective actions.

RECOMMENDATIONS

1. **The laboratory does not perform percent moisture determinations on sediment**

samples prior to extraction. The laboratory currently does not adjust extraction or concentration procedures based on > 70% moisture. The laboratory was told of the Region I percent moisture data validation policy that states: data for samples with > 70% moisture are estimated and data for samples with > 90% moisture are rejected. It was recommended that they address the possibility of adjusting the extraction procedures based on the required quantitation limits for their clients doing work in Region 1.

CONCLUSIONS

Premier Laboratory should be capable of producing aqueous and soil PCB data with sufficient, documented quality to support decision-making.

Should you have any questions, please do not hesitate to contact Ann Jefferies at 617-918-8373 or Nora Conlon at 617-918-8335.

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LABORATORY TECHNICAL SYSTEMS AUDIT

Laboratory: Premier Laboratory, LLC
61 Louisa Viens Drive
Dayville, CT 06241

Lab Contact: Bob Stevenson, QA Officer
Telephone: 860-774-6814
Fax: 860-774-2689

Programs: TSCA projects and RCRA Corrective Action for Pratt and Whitney in East Hartford, CT

EPA Requestor: Kimberly Tisa

Type of Evaluation: Technical Systems Audit - PCB analyses

Date of Evaluation: June 18, 2002

Laboratory Personnel:

Name	Title
Ron Warila	Laboratory Director
Bob Stevenson	QA/QC Manager
Philip Rusconi	CEO
Robert Laferriere	General Manager
Lab personnel noted inside questionnaire	

U.S. EPA Region I Evaluation Team:

Name	Title
Nora Conlon	QA Chemist
Ann Jefferies	QA Chemist

LABORATORY EVALUATION SUMMARY

I. SAMPLE RECEIPT, STORAGE, AND LOG-IN

Standard Lab turnaround: 7days

Describe the sample receipt, storage and log-in procedures.
{list sample custodian, SOPs, etc.} Sample custodians: Victor LeClerc - 8months
Bill Mallory - 1.5 years

Put samples on counter when they arrive, check Chain-of-Custody information, temperature of samples taken with IR temperature that has been calibrated against a NIST thermometer which is checked annually. Problems are documented and given to the Project Manager for contacting the client.

Custodians log-in samples into the lab designed LIMS, the program assigns project number (year month project number)
After Log-in, Project Manager checks paperwork, resolves problems with clients - paperwork kept with Project Manager, Labs are notified through computer backlogs and morning meetings.

Samples are stored in assigned refrigerators for 30 days (but really closer to 6 weeks) The UTC (client) PCB samples are separated. Solid waste is hauled by certified waste hauler, glass containers are crushed, plastic containers are recycled
Refrigerator temperatures are checked twice daily 1-4.5°C range - Bill adjusts for problems.

Things to check:
Check temperature logbooks, frequency of temperature checks, CA for temperature excursions for refrigerators.
What is checked when the samples arrive? (preservation, sample bottle condition and ID crossreferences) How is it documented?
How long are samples stored? minimum 30 days, refrigerated
Are chain-of-custody forms being used? yes
What internal chain-of-custody documentation is used? Logbook
What information is generated from login, who reviews it and what paperwork is given to the laboratories?
↳ Labs only get electronic "paperwork".

List Applicable Sample Receipt Logbooks. Check logbooks for evidentiary criteria such as signatures or initials, single-line crossouts, dates, secondary review, etc.
Book of Client Services SOPs

Logbooks: Temperature Logs; Waste Log; Internal Sample Custody Logbook - traces who removes and returns samples.

II. PCBs

Personnel(names, positions, training and experience):
Rich Warila - Organics Manager
Scott Lamitie
Amanda Swider

Preparation and Cleanup

Describe how soil samples are prepped.[Pressurized Fluid Extraction Method 3545, Rev. 1, October 19, 2001] [Sulfuric Acid Cleanup for PCBs, Method 3665A, Rev.1.1, October 19, 2001; Sulfur Cleanup, Method 3660B, Rev. 1.1, October 19, 2001] UTC samples - 24 hour turnaround.
(Things to consider: weighing samples and records; % moisture determination - adjustments for high % moisture; surrogate spiking; concentration technique; acid cleaning procedures; sulfur cleanup) Work list - check with log-in - morning meetings to notify that samples have arrived
Standing water decanted. Either 30g or 10g of soil are homogenized in a disposable "Whirlpak"; glassware & vessels are prepared. Samples are weighed and mixed with diatomaceous earth (DE) then placed in the Accelerated Solvent Extraction (ASE) vessel. Positions are tracked in ASE logbook. Each batch includes DE blank, LCS & project-specific MS/MSD of AR1254 or AR1660. Extract (30 minutes) is filtered through Na_2SO_4, Turbovap used - 42°C f.v. 20mL → 1.0mL aliquot acid cleaned - sulfur cleanup with Cu as needed.
Hexane only solvent for solid samples
Region I Data Validation policy for low % solids was described to the lab.
→ Do not currently measure % moisture first - do not have procedures for adjusting sample size for high moisture

Describe how aqueous samples are prepped.[Separatory Funnel Liquid-Liquid Extraction, Method 3510C, Rev. 1.2, April 3, 2002]
(Things to consider: recording sample volume, pH adjustment, surrogate spiking, concentration technique, acid cleaning procedures, sulfur cleanup) Sep funnel (Plastic) - use $MeCl_2$, check pH of samples and adjust if necessary. QC samples Blank, LCS, MS/MSD - use AR1660 (1mL of 4 ppm solution) Entire sample is used - mark volume, measure with graduated cylinder. Add 60mL $MeCl_2$, shake 2 aliquots, 3 times, drain solvent layer through Na_2SO_4 filter, Rotodown by Turbovap to 2mL, exchange to hexane - send 1.0mL to instrument lab, archive 1.0mL. Waters are not routinely cleaned.

Calibration, Surrogates and Matrix Spike Standards Prep:

Check stock solution concentrations and the measurement of volumes. Write down the concentration levels. 1. How many standard solutions are used and what compounds are in each one? 2. Are standards traceable? 3. How are the standards documented? 4. Is there a second source? Acceptance limits and corrective action. Where are standard preparations recorded? [Single point AR1221, AR1232, AR1242, AR1248, AR1254; multipoint AR1660 (0.2, 1.0, 2.0, 5.0, 10.0 mg/L); Reporting limit 0.40 µg/L waters; 13.3 µg/kg soils for each AR] logbooks right levels also run a 0.1 sometimes.

Standard prep documentation.
① Purchased Standards Receipt Logbook - track vendor lot - given unique number
② Stock Standard Preparation Logbook - given stock number, expiration date on bottle
③ Working Standard Prep Logbook - do QC on working standard - QC notebook

Use Absolute standards for calibration standards
Use Accustandard for spikes for 2nd source.
Surrogates: DBC & TCMX 1mL of 4ppm standard → final concentration in instrument 2ppm.

II. PCBs (continued)

Sample analysis [Polychlorinated Biphenyls by Gas Chromatography SW 846 8082, Rev. 1.1, March 12, 2002] Analyst: Weison Huang – 2 years in the lab

Record analytical sequence for initial cal to end of a sequence. Things to check: concentration levels in initial calibration; blank frequency; injections between calibrations, continuing calibration concentration, analytes and frequency; dilutions; Aroclor identification

Analyst receives extraction list – copy from Extraction Logbook. Analyst runs 6pt curve but only uses 5 pts – evaluate by % RSD based on area – criteria < 20% RSD each evaluated peak in each column. Surrogates – if >20% RSD can evaluate by linear regression r2 > 0.990 but rarely used, single AR at 2ppm

Sequence: ICAL (multi-point AR1660), single AR, IBLK, MBLK, LCS, samples (up to 10) CC (2ppm AR1660, AR1248, AR1254), IBLK, 10 more, end with AR1660 + Target AR. If dilution required aim for mid curve.

Quality Control Samples: acceptance limits and associated corrective action - **[specific QC criteria are not listed in the SOP]** Criteria developed for each peak by control chart,

Method Blanks[< quant limit for project](must have nothing that interferes with identification and quantitation) IBLK < Detection Limit

Method Blanks – surrogates must meet on both columns – one reinject, still out – reextract,

Surrogates [TCMX, DCB] Corrective Actions? Control chart limits set every 6 months.

Spiked samples (LCS, MS/MSDs?) What analytes are in the spike solution? What source, same as calibration? What is the frequency for spiked samples? Corrective Actions? received copy of acceptance windows.

LCS – evaluate, if out, reinject – if still out reextract the whole batch

MS/MSD – if LCS ok and surrogates ok → it's ok – fill out nonconformance for AR1254: 50% RPD / 45 – 145% window

Data Review

How are compounds qualitatively identified? How are retention times evaluated? Are pattern recognitions used for identification? Are there methods to determine if there are any interfering non-target compounds? What criteria are used?

Qualitative – overlap with standard – RT updated every 72 hours.

If there are interferences will send back to extraction lab for cleanup.

Get's complicated with multiple ARs in a single sample.

How are target compounds quantitated? Peak heights or areas? How are the peaks summed (3 to 5 peaks) how are they selected? From initial calibration curve? External standard method (CF)? Area counts plugged into the regression? Continuing calibrations evaluation [85 -115 % R, frequency, beginning, every 10 recommended, end of sequence]

[Ask for a quantitation demonstration.]

If ICAL evaluated by % RSD, then use CF AR1016 + AR1260. If other AR, use nearest single point 'injection' criteria for continuing – ±15% of expected concentration.

If linear regression, quant by curvefit.

II. PCBs (continued)

What are the review procedures? Is there secondary review?
There are multiple levels of documented review-

How are the data reported? How are specific client requirements communicated?
Data are transfered directly from the instrument to the target software
Analyst sends data over, Supervisor reviews 100% of data
Comments for nonconformances are incorporated. Hardcopy - manually integrations
initialed and dated, Special requirements documented on Worklist

When were the most current MDL studies performed and are they available?
Frequency of computer data back-up? - daily - tapes taken home by administrator Paul Milne
List instrumentation HP5890 - dual column, single Y split injector
Dual injector, dual column, dual detector?
Columns: RTX-CLP Pesticide I 30m 0.32mmID 0.50μm film thickness
RTX-CLP Pesticide II 30m 0.32mmID 0.25μm film thickness.
How are instruments maintained? Are there sufficient replacement parts?
Done in house
have a local contractor for big problems

Are SOPs readily available? - yes - electronic versions

List Logbooks: Check for evidentiary criteria such as signatures or initials, single-line crossouts, dates, secondary review, etc.
- Run Log; Maintenance Log
Extraction Log from sample prep.

Data Package - raw data include - electronic deliverables in 24 hours
Client can validate in ~2 hours

Forms data
2 surrogates
3 MS/MSD
3 LCS
4 Blank Summary
6 ICAL
7 CCAL

Changes to data are color-coded in the LIMS. Makes it very easy to track.

III. OTHER SYSTEMS AND DOCUMENTATION

(Quality Manual, Revision 2.3, Effective Date: March 1, 2002)

Describe how staff are trained on the Quality Assurance Program. – New employees
Chemical Hygiene Program first
Read Quality Manual – Sign Ethics statement
Training with SOPs and on-the-job
Do initial demonstration of capabilities which is kept on file.

What is the frequency of internal audits? How frequently are you audited by outside organizations? What certifications do you maintain?
Internal audits – continuous – rotate through labs.
Do double blind proficiency tests occasionally.

Certifications: NELAC, CT, MA, RI, ME, NH, NJ

What is the procedures for handling QA/QC problems? Are there forms, how are problems tracked for identifying systematic problems?
Nonconformance forms – electronic versions
Bob (QA/QC Manager) receives all hardcopies

Other Stuff:
Health and Safety information? – Chemical Hygiene Plan
Waste Disposal? – Color coded (not observed)
Recently audited by CT DEP – passed with flying colors.

Other: SOPs filed electronically to control changes.